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(54) METHOD AND APPARATUS FOR THE CONTINUOUS PRODUCTION OF COLLAGEN FILMS AND THE CONTINUOUS TREATMENT OF FILMS SO PRODUCED

(71) We, CENTRE TECHNIQUE DU CUIR, SOCIÉTÉ ANONYME, a French Body Corporate, of 181 Avenue Jean-Jaurès, Lyon 7ème, Rhône, France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed. to be particularly described in and by the following statement:—

The present invention relates to a method and apparatus for the continuous production of collagen films and the continuous treatment of films so produced. More particularly it relates to a method and an apparatus for the preparation of collagen films, sheets, and other similar products, constituted of collagen, either alone, or in combination with woven or non-woven fabrics, natural, artificial or synthetic, as well as their subsequent treatment with a view to numerous and varied applications.

Starting from suspensions, dispersions or from gels of collagen in water or in an organic solvent, it is possible to obtain objects of collagen, such as filaments especially surgical sutures, films, sheets and envelopes for foodstuffs. The recognised techniques are numerous and they call upon, in general, the extrusion of collagen from its dispersions or its aqueous gels in the form desired, and its coagulation by chemical agents or physical means. Sometimes, the use of dispersions or gels of collagen in an organic solvent has been suggested, for in this case, the solvent enables the bacterial contaminations observed in aqueous media to be avoided and the later operations of drying the collagenic articles to be facilitated. In addition,. the mechanical properties of the collagen in the form of filaments, films or of sheets are relatively weak as regards resistance to traction, breaking elongation or tear resistance, and this explains the numerous difficulties encountered in obtaining objects of collagen of large size.

It is well known that collagen may be placed in suspension in water at a pH in the neighbourhood of the "isoelectric point", that is to say at pH values within the range 5 and 6.5. Its solubilisation or its dispersion in water or in an organic solvent cannot equally be effected if its electrical state is modified by increasing the number of positive or negative electrical charges, and this is generally obtained by the addition of an acid or basic, inorganic or organic chemical agent.

The natural fibrous properties of the collagen as well as its considerable film-forming power make it possible, as for numerous natural proteins and synthetic polymers, to reobtain it from its solutions or from its dispersions in the form of filaments, fibres and films. In all cases, however, it is necessary to coagulate the protein and especially collagen. The obtaining of threads and of filaments is done in most cases by the wet method by extruding a collagenic dispersions or gel, in aqueous acid medium, into coagulant baths constituted of organic solvents like methanol, or acetone, and including inorganic bases like ammonia, soda, or organic like amines.

Objects of collagen usable as food packages, especially sausage envelopes, are obtained by extrusion of a tube of inflated collagen and extended on the inside by air under slight pressure and by immediate coagulation in solutions or mineral salts at high concentration like sodium chloride, sodium sulphate or ammonium sulphate.

Sponges and articles of spongy character are prepared by using techniques of drying in the cold under vacuum, like lyophilisation, which enable, whilst coagulating the collagen by the previously mentioned means. its dehydration and the creation of air zones included in the coagulated mass.

In the same way, films of collagen can be obtained by extrusion of dispersions or of gels on to a hydrophobic support, and, either dried under a current of air, or coagulated in the

organic or inorganic baths previously mentioned. The latter method of production is generally discontinuous and used for laboratory preparations, when collagen is used alone.

In all cases, however, it is necessary, to obtain good coagulation, to neutralise the acid or basic chemical agent which has served for the solubilisation or for the dispersion of the collagen in order to bring the latter towards its isoelectric point, that is to say, of minimum swelling and of maximum insolubilisation in water. In addition, its strong capacity for absorption of water and water vapour necessitates very often the subsequent action of tanning agents or of cross-linking agents which render it rot-proof and which confer on it greater heat stability. These various operations of use and of transformation are in general delicate and are carried out, except for obtaining a thread, with a certain discontinuity necessitating numerous controls and treatments which greatly reduce their profitability.

It is an object of the present invention to overcome these drawbacks.

To this end, it relates to a method and an apparatus automated for the continuous production of collagen films, sheets, and other similar products constituted of collagen, either alone, or in combination with woven or non-woven fabrics, natural, artificial or synthetic, as well as their subsequent treatment with a view to numerous and varied applications.

According to the present invention, said method comprises preparing, in a reactor operable under vacuum or under pressure, a solution, a dispersion or a gel of collagen in water or in an organic solvent, by adding with agitation a solution of an acid or of an inorganic or an organic alkaline compound, to swell the collagen and impart to the dispersion or solution a desired viscosity, the starting material being constituted by wet or dry collagen fibres, regenerated or taken in the native state; extruding under pressure the collagen solution or dispersion after being deaerated or aerated in the reactor onto an endless travelling belt or band of a hydrophobic polymeric material functioning as a casting surface by means of a slot die, and drying the film at a temperature within the range 30 and 40°C, without causing degradation or hydrolysis of the collagen, to obtain a collagen film for applications such as surgical films, external dressings, packages for foods or chemical products, semi-permeable membranes, sponges for surgery, filters for cigarettes, materials for shoes and fancy leather goods and other similar products; stripping the collagen films from the endless belt or band, and winding it under constant predetermined tension onto spools to form packages of desired size and weight.

These various operations may be carried out automatically either successively, or simultaneously, in one or several installations, in a relatively short time, and with increased profitability, the width of the film of collagen or of the impregnated textile material being variable according to the article desired.

Naturally, whilst the collagen dispersions are still in the reactor, that is to say before they have been extruded, the chemical agents necessary for the subsequent modifications of the collagen and corresponding to the desired characteristics of the finished product, such as colouring materials, fatty materials, tanning agents, antiseptics, plasticisers, and natural or synthetic resins, can be introduced into the said reactor.

In certain cases, and especially if a better constructed collagen film is desired, according to a particular form of practising this method, the collagen film obtained by extrusion may be coagulated, before drying, by means of liquid or gaseous chemical agents.

For example, this coagulation can be effected by passage of the film through coagulating baths consisting of solutions of inorganic alkaline compounds such as ammonia or organic bases such as amine in volatile dehydrating organic solvents such as methanol, ethanol, acetone or the like.

According to a variation of this method, the film is coagulated before drying by means of a volatile alkaline compound such as gaseous ammonia.

Where it is desired that the film be reinforced with a fibrous support, its extrusion on the endless travelling belt or band is effected either upstream or downstream of the deposition on the same travelling belt of a web of a woven or non-woven textile fabric, which is carried along by the travelling belt at the same speed as the collagen film.

Preferably, in the case where the collagen film is extruded downstream of the deposition of the web of textile fabric, the impregnation of this latter by the collagen material is improved by passage of the fabric web above an aspiration zone situated downstream of the extrusion point.

The apparatus according to the invention comprises a dispersion reactor operable under vacuum or under pressure, connected to storage vats by a system of change-over valves, a pumping system adapted for the precise regulation of the flow of the solution, the dispersion or the gel of collagen through a slot die, situated at the end of a metallic frame bearing a casting surface constituted by an endless travelling belt of hydrophobic material and driven at a constant speed by a motor provided with means for adjusting its rate of rotation to any desired constant value, one or more drying tunnels whose temperature is kept constant by thermostatic control and whose atmosphere is renewed by an assembly of tangential ventillators, and which are traversed by at least one of the runs of

the aforesaid travelling belt, a device for continuously stripping the dried film web from the travelling belt and winding the collagen film or the web of textile fabric impregnated with collagen onto a spool in the zone where it is stripped from the belt.

Advantageously, to enable the treatment of the film of collagen or of collagen-impregnated textile fabric by this apparatus, the latter is associated an apparatus which comprises a metallic frame supporting successively a series of vats between which are included suitable baths and in which the film web is looped by turning and guiding rollers. a drying tunnel and a device for winding the film web onto a spool at a constant tension.

In order that the invention may be more fully understood one embodiment of an apparatus for practising the method according to the invention and several embodiments of the method are described below purely by way of illustrative and non-limiting examples, with reference to the accompanying diagrammatic drawings in which:

Figure 1 is a diagrammatic view of the apparatus used for the formation of the collagen film and of the impregnated textile fabric;

Figure 2 is a diagrammatic view of a drying tunnel;

Figures 3, 4 and 5 are diagrammatic views of a system for detaching and winding the collagen materials; and

Figure 6 is a view of the apparatus serving for various treatments of the collagen.

Figure 1 shows diagrammatically an apparatus according to the invention. This apparatus comprises a dispersion reactor - enabling the preparation of solutions, dispersions or gels of collagen, having a concentration within the range 1 and 5% in an aqueous medium, and between 1 and 15% in an organic solvent. The introduction or elimination of air bubbles can be carried out easily in the reactor 1 as well as the addition of various chemical agents compatible with the collagen and the dispersing medium, such as colouring agents, tanning agents, fatty materials, antiseptics, plasticisers, natural or synthetic resins, and in a general manner, any substances useful for the applications contemplated.

The dispersions are stored in vats 2 which are alternately being filled and being used and in which the dispersions may undergo maturing for several hours. The vats 2 are produced of a non-corrodable material which is resistant to pressure, such as stainless steel, synthetic polymers, or any other similar material having the preceding characteristics.

The dispersions are conveyed from the storage vats 2 to a slot die 5 by means of a pumping system 3 comprising a gear pump of stainless steel, of conventional type, driven by a motor variator and protected by a torque limiter.

Filters 4 of non-corrodable materials and capable of withstanding high pressures are placed, firstly, between the reactor 1 and the storage vats 2 to eliminate from the dispersion non-dissolved materials and to ensure the protection of the pump 3 and, secondly, between the pumping system 3 and the die 5 in order to retain the last impurities capable of partially obstructing the die 5.

Each filter 4 has a filtration surface of around 2 sq. dm. and encloses a stainless grill of mesh 0.5 to 1 mm side. The purification of the dispersions can be carried out by a single filter or by several filters mounted, either in series, or in parallel, but their operation is for several hours without observing considerable clogging.

The die 5 is of the slot type, the slot being wider than it is deep. Its dimensions can vary within wide limits according to the applications contemplated but in a general manner, its length must be distinctly greater than its width with the purpose of ensuring a homogeneous distribution of the dispersion. The die 5 is produced of a non-corrodable material, like stainless steel, superpolyamide, poly - tetrafluoroethylene, or any other similar material, and it is provided with adjustable lips enabling the distance between them to be varied up to 5 mm, this latter being determined by keying elements of flexible material which also have the purpose of conducting the collagen dispersion on to an endless travelling belt 7 avoiding any necking of the collagen layer.

The die 5 deposits the layer of collagen in dispersion on the travelling belt 7 constituted by a travelling endless band of hydrophobic material such polyethylene, poly - propylene, polybutene, polyvinyl chloride, or any other similar material.

Figure 3 shows a method of fixing the travelling belt 7; the latter is fixed on each side of two chain conveyors 19, provided with fixing lugs 20, by means of springs 21 which are connected to the travelling belt 7 by eyelets 22. The chain conveyors 19 are held and guided by rails and shafts provided with pinions and the driving of the belt 7 is ensured by a variable speed motor 8 at a constant speed but adjustable according to the applications contemplated. The driving shaft of the variable motor 8 is placed slightly downstream of the die 5 so as to obtain a regular passage of the belt 7 on the spreading of the layer of collagen.

As shown in Figure 1, the apparatus comprises, moreover advantageously, a device enabling the impregnation or the coating of textile materials, like gauzes fabrics and non-woven textile fabrics. A bobbin winder 9a, placed upstream. or 9b placed downstream of the die 5 effects the deposition either below or above the layer of dispersed collagen, extruded by the die 5, of the woven or non-

woven fabric. When the bobbin 9a is used, the impregnation in thickness of the textile fabric which proceeds can be obtained by reduced pressure furnished by means of a vacuum chamber 10. The belt 7 carries the layer of collagen obtained by means of the die 5 towards drying tunnels 11 and 11a where the solvent or the dispersion medium is evaporated, whether this is water or an organic solvent. Nevertheless, in a general manner, the collagen is coagulated in the form of fibrils or of fibres in the midst of the film by deflation and neutralisation of the latter by means of an alkaline agent, a saline solution, or a solvent with a strong affinity for water containing an alkaline agent.

In a preferred embodiment of the invention, the coagulation of the collagen is effected by means of ammonia gas which is led close to the surface of the layer of gel by a nozzle 6 of elongated shape. The layer of gel becomes opaque and whitish by the formation of collagen fibrils.

A method variation consists in passing the belt 7 and the layer of gel into a dehydration solvent of low boiling point like acetone or methanol, capable of containing an alkaline, inorganic, or organic agent, permitting neutralisation and coagulation of the collagen. The layer or gel traverses one or several vats containing solvent (which are not shown in the drawing) and the dehydration as well as the neutralisation can be controlled by density measurements and by automatic potentiometer determinations.

The transformation of the layer of gel into a film is effected in the drying tunnels 11 and 11a. Figure 2 shows diagrammatically the first tunnel 11. In this apparatus, the belt 7 is supported so as to eliminate any accumulation of heat capable of transforming the collagen into gelatine. For this same reason, the drying temperature must be within the range 30 and 40°C and in the tunnel 11, where the water content of the gel 14 is considerable, the temperature is held constant by means of thermostatic control piloted by a platinum resistance probe 18 placed in contact with the belt 7. In the other drying tunnel 11a, such regulation is not essential.

Heating is effected by conventional techniques comprising hot air, infra-red radiation, hot plate, or any other similar means, but in a preferred embodiment of the invention infra-red radiation is used, being more flexible in use and having the advantage of minimum energy consumption. Heating is thus effected by a series of infra-red tubes 15 arranged at about 20 cm from one another, above the layer of gel 14 at a distance not exceeding 20 cm, and it is coupled with vigorous ventilation ensured by regularly spaced tangential ventilators 16. The ventilation is necessary since it enables the maximum evaporation of water whilst cooling the layer of gel 14. Moreover, the evacuation of the moist air is ensured by aspirating turbines 17. More intense drying of the film increases the flexibility and raises its shrinkage tension on the support with, as a possible result, premature detachment of the film, and, in the limit, its rupture. Also, it is desirable to conserve in the film a water content within the range 5 and 20%, and this can be additionally obtained by continuous conditioning of the film at the end of drying in an atmosphere having, at ambient temperature, a relative humidity within the range 40 and 60%.

After drying in the tunnels 11 and 11a, the film or the textile material impregnated with collagen is detached from the belt 7 by a device 12. Figure 3 shows a diagrammatic view of this device; the latter is principally constituted by a three-toothed blade 23 which is interposed between the belt 7 and the film 25 and of which the shape ensures a detachment profile 24 of maximum length, thus reducing the risks of folds and tears. The adhesivity of the film 25 on the belt 7, and consequently its detachment depends on the nature of the film, the method of coagulation, the nature and the proportion of chemical agents added like plasticisers, fatty materials, natural or synthetic resins. When this adhesivity is high, which is the case for the film obtained from air-filled dispersions, it is necessary to replace the trident blade 23 by a rotary rod. Figure 4 shows a diagrammatic view of this device; it is simply composed of a rod 26, driven by a slow movement, actuated by an adjustable speed motor, and inserted between the film 25 and the belt 7.

After detachment, the film or the textile material impregnated with collagen is wound onto a spool. Figure 5 shows in more detail the spooling system 13. It comprises a receiving spool 27, driven by an electromagnetic torque reducer motor assembly 28 and a moving element 29, arranged between two loose rollers 32 and ensuring constant tension of the winding of the film 25 on the spool 27. Moving element 29 is provided at its lower end with an optical wedge 30 which acts on a photo-electric system 31 connected to the electromagnetic coupler 28.

The incorporation of foreign chemical substances cannot often be effected directly in dispersion or gels of collagen by reason of their insolubility in water or organic solvents, or their incompatibility with collagen which can then transform or coagulate it. Moreover, the treatment of a film adherent on a support presents numerous difficulties as a result of the very slow diffusion through the film through a single face and by reason of the dangers of corrosion of the material. Also, it is often necessary to subsequently treat the film of collagen or of impregnated textile material. The various treatments applic-

able, either separately or simultaneously, to the collagen, comprise cross-linking by inorganic or organic tanning agents, dyeing, plasticising by polyols, fatty materials, or any other similar plasticiser, waterproofing, the introduction of antiseptic or therapeutic agents, and in a general way, all operations corresponding to the desired articles.

Figure 6 shows a diagrammatic view of the apparatus used for the treatment of the film or of the material impregnated with collagen. This apparatus comprises a spool winding machine 33, delivering the film or the material impregnated with collagen, which is taken under load by unwinding rollers 34 and sent into a succession of vats 35 where the collagen undergoes the desired treatments. Passage from one vat to the other is ensured by driving rollers 36 and, after the last treatment, the material is led into a drying tunnel 37 fed with warm air at constant temperature by a system of tangential ventilators 38 and in which it can undergo a certain stretching by simple action on the speeds of the driving rollers 39 installed in the tunnel 37. The dried material is wound under constant tension on a receiving spool 41 identical with the spool 27 of Figure 5, by means of a moving element 40, similar to the assembly 29 of this same figure.

In a variation of the method, the film can be treated immediately after its detachment and, in this case, the treatment apparatus is connected directly to the film production installation. The film or the collagen-impregnated textile material is then detached from the belt 7 by the assembly 12 and is sent into the treatment vats 35 by means of the winding rollers 34.

The invention is illustrated by the following non-limiting examples:

EXAMPLE 1

A 2% dispersion of collagen is prepared in the following way: a suspension of 300 grammes of dry collagen fibres in 10 litres of water is introduced into the reactor and stirred under vacuum for 5 minutes, to eliminate air bubbles. A solution containing 75 grammes of lactic acid and 90 grammes of glycerol in 4.535 litres of water is then added and the whole stirred under vacuum for 10 minutes.

The homogeneous and air-free dispersion thus obtained is filtered under pressure on a stainless grid of mesh 0.5 mm side and stored for 24 hours in a vat; it is then sent under air pressure of 1 kg/cm2, from the vat towards the gear pump which, at the speed of 7 revolutions/minute, ensures a constant flow of 4 litres/hour. The gel is spread on the travelling belt in a homogeneous thin layer by the die of which the distance between the lips is adjusted to 1.5 mm. The collagen is soon coagulated by the gaseous ammonia of which the flow is about 2 litres/minutes. The speed of advance of the travelling belt was fixed in this laboratory trial at 10 metres/hour.

The heating power of the upper tunnel is 300 watts per metre and that of the lower tunnel 450 watts per metre, the length of each of them for this preparation being fixed at 16 metres.

The film is perfectly dry in the middle of the lower tunnel and it is then easily detached by the trident blade then wound under constant tension of 200 grammes. The film obtained is in the form of a translucent film having a homogeneous thickness of 0.03 mm and a density in the neighbourhood of 1.3.

The mechanical properties of this collagen film tested on an Instron dynamometer are as follows:

Tensile strength	2.4 kg/mm2
Elongation at rupture	54%
Initiated tear resistance	0.9 kg/mm

These properties are a little weaker in the transverse than in the longitudinal direction, and the observed anisotropy is explained by the preferential direction of flow of the gel on the travelling belt and by the differences in shrinkage tension in the course of drying.

EXAMPLE 2

A 2% dispersion of collagen is prepared in the reactor, by agitation, under air pressure of 1 kg/cm², of a suspension of 300 grammes of dry collagen fibres in 10 litres of water. After 5 minutes stirring, a solution containing 75 grammes of lactic acid and 30 grammes of glycerol in 4.535 litres of water is added, and the mixture is produced by stirring under air pressure of 1 kg/cm² for 10 minutes. The homogeneous and airfilled stored for 24 hours in a vat whence it is dispersion is filtered under pressure and driven under air pressure of 1 kg/cm² towards the gear pump which, at the speed of 7 turns/minute ensures a constant flow of 4 litres/hour. The gel is spread on the travelling belt in a homogeneous thin layer by the die of which the distance between the lips is adjusted to 1.5 mm. The collagen is soon coagulated by a current of ammonia gas of which the flow is about 2 litres/minute. The speed of advance of the travelling belt is fixed for this production at 10 metres/hour.

The collagen film is perfectly dry at the middle of the lower drying tunnel of which the drying power is adjusted to 450 watts per metre whilst that of the upper tunnel is fixed at 300 watts per metre. It is detached from the travelling belt by the rotary bar system and a part is wound under constant tension of 200 grammes. The film thus ob-

tained has the form of an aerated opaque pellicule having an average thickness of 0.2 mm and a density of 0.25.

Its mechanical properties tested on an Instron dynamometer are as follows:

Tensile strength	0.5 kg/mm2
Elongation at rupture	15%
Initiated tear resistance	0.3 kg/mm

The second part of the dried and detached film is sent directly on to the treatment installation comprising four vats of 5 metres in length containing respectively a solution of complex sulphate of chromium at 50 grams per litre, of 45° Schorlemmer basicity, a solution of acid sodium carbonate at 50 grams per litre, of pure water for rinsing and circulating continuously at slight flow, and an aqueous solution of glycerol at 100 grams per litre. The film passes successively into these baths at the same speed of 10 metres per hour, and it is dried between each vat by driving rollers. It is finally dried in the latter tunnel at 50°C without undergoing stretching and wound under constant tension of 200 grams.

The film obtained is slightly tinted pale blue, very flexible, and includes 2% of chromium expressed as Cr_2O_3 as well as 30% of glycerol. Its shrinkage temperature is 77° C and its mechanical properties are appreciably close to those of the same untreated film.

Example 3

A dispersion of collagen free of air bubbles of composition identical with those of the preceding examples and prepared in the same manner is spread, by means of the gear pump and the die, on a continuous web of non-woven textile fabric constituted by fibres of superpolyamide and of cellulose acetate and drawn by the travelling belt from its spool unwinder. The flow of the die is adjusted so that the web of non-woven textile fabric is entirely impregnated, but without excess, by the collagen gel, after passage over a vacuum chamber where these exists a reduced pressure in the neighbourhood of 0.5 bar.

The collagen is soon coagulated by a current of ammonia gas of which the flow is about 2.5 litres/minute. The speed of advance of the travelling belt is fixed for this operation at 10 metres/hour. The material is practically dry at the middle of the lower tunnel and it is detached from the mat by the rotary bar system, then wound under a constant tension of 300 grams.

The material obtained has lost about 60% of its initial thickness and it is possible to distinguish under the microscope the fibrils of collagen between the constituent fibres of the initial mat.

WHAT WE CLAIM IS:—

1. A method for the continuous production of collagen films and the continuous treatment of films so produced comprising preparing, in a reactor operable under vacuum or under pressure, a solution, dispersion or a gel of collagen in water or in an organic solvent, by adding with agitation a solution of an acid or of an inorganic or an organic alkaline compound, to swell the collagen and impart to the dispersion or solution a desired viscosity, the starting material being constituted by wet or dry collagen fibres, regenerated or in the native state; extruding under pressure the collagen solution or dispersion after being deaerated or aerated in the reactor onto an endless travelling belt or band of a hydrophobic polymeric material functioning as a casting surface by means of a slot die, drying the film at a temperature within the range 30 and 40°C, without causing degradation or hydrolysis of the collagen, to obtain a collagen film for applications such as surgical films, external dressings, packages for foods or chemical products, semi-permeable membranes, sponges for surgery, filters for cigarettes, materials for shoes and fancy leather goods and other similar products; stripping the collagen film from the endless belt or band, and winding it under constant predetermined tension onto spools to form packages of desired size and weight.

2. A method as claimed in claim 1, wherein chemical agents necessary for the subsequent modifications of the collagen and corresponding to the characteristics of the desired product such as colouring materials, fatty materials, tanning agents, antiseptics, plasticisers and natural or synthetic resins, are introduced into the said reactor.

3. A method as claimed in claim 1 or 2, wherein the film of collagen obtained by extrusion is coagulated, before drying, by means of liquid or gaseous chemical agents.

4. A method as claimed in claim 3, wherein coagulation is effected by passage of the film through coagulant baths consisting of solutions of inorganic alkaline components such as ammonia or organic bases such as amine in volatile dehydrating organic solvents Such as methanol, ethanol, acetone or the like.

5. A method as claimed in claim 3, wherein the film is coagulated before drying by means of a volatile alkaline compound such as gaseous ammonia.

6. A method as claimed in claim 1, wherein the extrusion of the collagen film on the endless travelling belt or band is effected on a web of a woven or non-woven textile fabric, which is carried along by the travelling belt at the same speed as the collagen film.

7. A method as claimed in claim 6, wherein the extrusion is effected upstream of the

deposition of the web of textile fabric on the travelling belt.

8. A method as claimed in claim 6, wherein the extrusion is effected downstream of the deposition of the web of textile fabric on the travelling belt.

9. A method as claimed in claim 8, wherein the impregnation of the fabric web by the collagen material is improved by passage of the fabric web above an aspiration zone situated downstream of the extrusion point.

10. An apparatus for practising the method as claimed in any one of claims 1 to 9, comprising a dispersion reactor operable under vacuum or under pressure, connected to storage vats by a system of changeover valves, a pumping system adapted for the precise regulation of the flow of the solution, the dispersion or the gel of collagen through a slot die, situated at the end of a metallic frame bearing a casting surface constituted by an endless travelling belt of hydrophobic material and driven at a constant speed by a motor provided with means for adjusting its rate of rotation to any desired constant value, one or more drying tunnels whose temperature is kept constant by thermostatic control and whose atmosphere is renewed by an assembly of tangential ventillators, and which are traversed by at least one of the runs of the aforesaid travelling belt, a device for continuously stripping the dried film web from the travelling belt and winding the collagen film or web of textile fabric impregnated with collagen onto a spool in the zone where it is stripped from the belt.

11. An apparatus as claimed in claim 10, associated with an apparatus which comprises a metallic frame supporting successively a series of vats between which are included suitable baths and in which the film web is looped by turning and guiding rollers, a drying tunnel and a device for winding the film web onto a spool at a constant tension.

12. A method for the continuous production of collagen films, and the continuous treatment of films so produced, substantially as hereinbefore described with reference to the Examples.

13. An apparatus for the continuous production of collagen films, and the continuous treatment of films so produced, substantially as hereinbefore described with reference to the accompanying drawings.

14. Collagenic products, whenever made from a film made by a method as claimed in any of claims 1 to 12.

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1234358 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

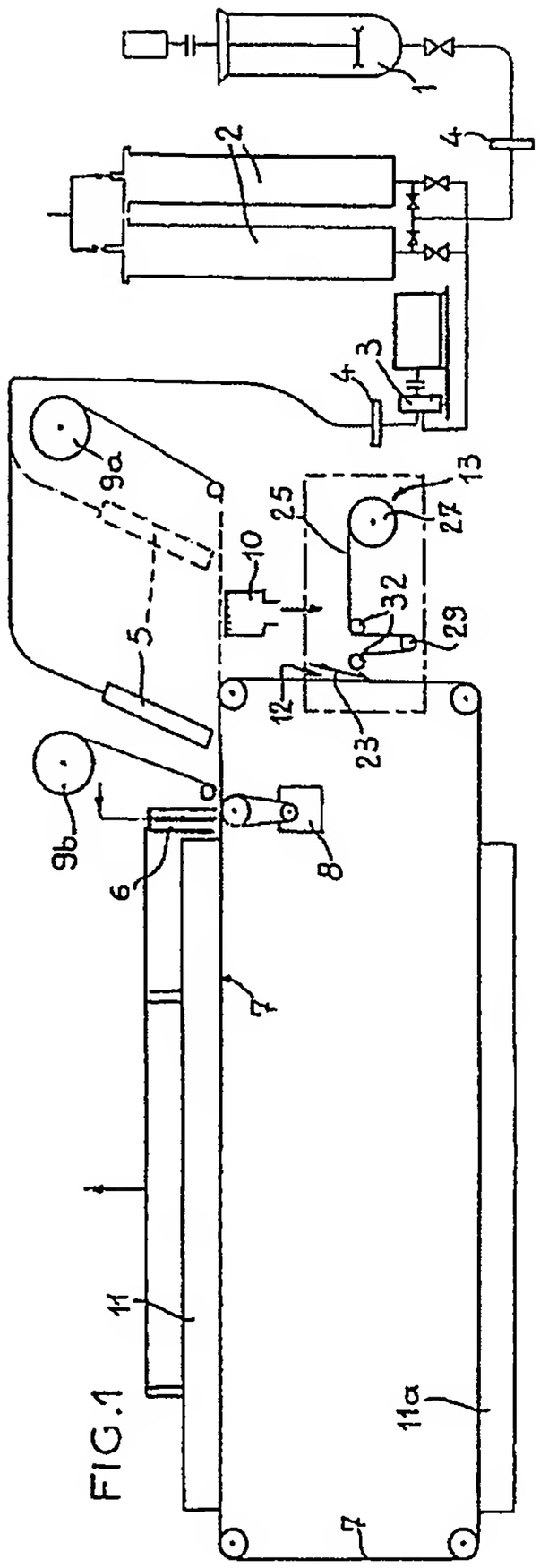

FIG.1

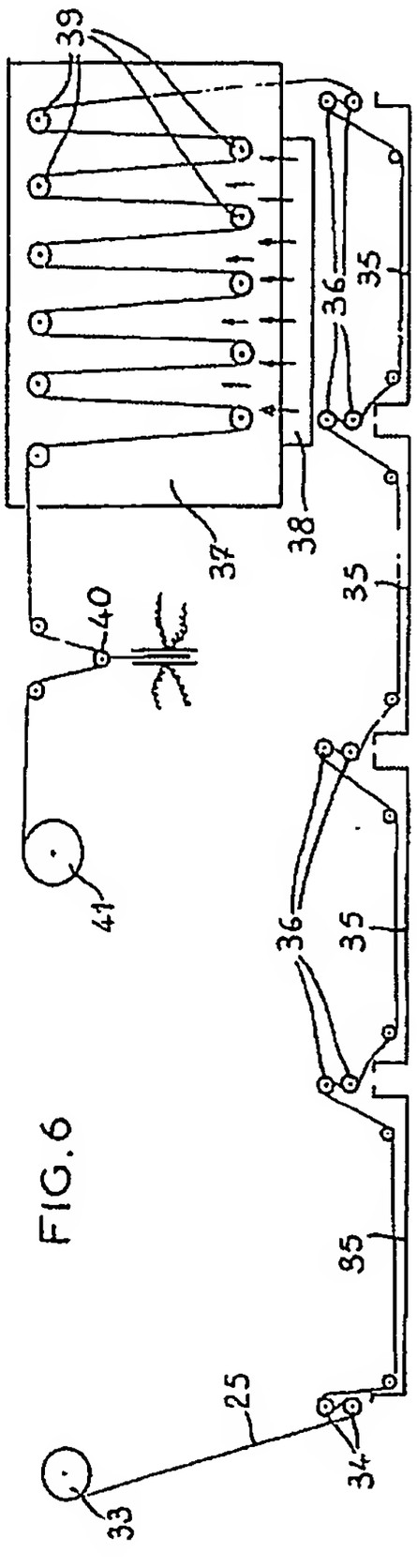

FIG.6

1234358 COMPLETE SPECIFICATION
2 SHEETS This drawing is a reproduction of the Original on a reduced scale
Sheet 2